

The Crystal Structure of a Sodium Complex, Sodium Perchlorate-Bis-[*NN'*-ethylenebis(salicylideneiminato)copper(II)]

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THE formation of oxygen-bridged bi- and trinuclear complexes from metal salicylaldimines is well established.¹⁻³ Tetradentate Schiff base (L) complexes of bivalent metals (M), ML, are reported^{1,2} to form compounds ML, MX₂ with transition-metal (M') halides, M'X₂, or trinuclear complexes [(LM)₂M']Y₂ with M'Y₂ (Y is a non-coordinating anion).

four sodium ions and four chlorine atoms in the unit all lie on crystallographic two-fold axes while the four xylene molecules are situated about centres of symmetry.

The environment of one sodium ion is depicted in the Figure. It is surrounded approximately octahedrally by oxygen atoms, two from a chelating perchlorate ion, Na...O(3) = 2.6 Å and two

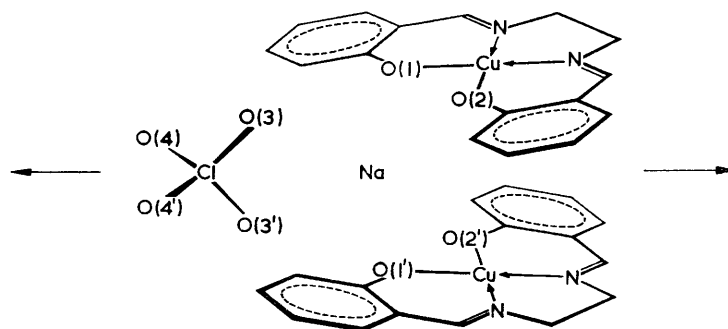


FIGURE. Diagram showing one molecule of sodium perchlorate-bis-[*NN'*-ethylenebis(salicylideneiminato)copper(II)] with the hydrogen atoms omitted. The crystallographic two-fold axis is indicated by an arrow.

Gruber⁴ described the formation of a maroon precipitate from a chloroform solution of the Schiff base complex, *NN'*-ethylenebis(salicylideneiminato)copper(II), by addition of sodium perchlorate in ethanol. Kolombos⁵ repeated the preparation and recrystallised the adduct from ethanol-*p*-xylene (1:1) giving pleochroic crystals containing, for each sodium perchlorate, two molecules of the Schiff base complex and one of *p*-xylene. We report its crystal structure analysis.

Crystal data. C₃₂H₂₈N₄O₄Cu₂·NaClO₄·C₈H₁₀. Monoclinic *a* = 24.463, *b* = 11.248, *c* = 14.785 Å, β = 101.03°, μ = 13.5 cm.⁻¹ (for Mo-K_α), D_m = 1.60, Z = 4, D_c = 1.49. Space group C2/c. Diffractometer measurements of intensities.

The structure was solved from 1083 unique reflections by three-dimensional Patterson and Fourier syntheses, and refined by block-diagonal least-squares to the present *R*-value of 0.075. The

from each of two Schiff base complex systems, Na...O = 2.3 Å. The whole entity shown in the Figure is neutral and makes only van der Waal's contacts with other such entities and with the xylene molecules in the unit cell. In particular there are no contacts less than 3.5 Å from the oxygen atoms to sodium ions. If the site occupied by sodium were occupied by a transition metal the resulting compound would undoubtedly be regarded as a trinuclear complex. The co-ordination about the copper is square, Cu-O = Cu-N = 1.9 Å and each Schiff base complex system is approximately planar.

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⁴ S. J. Gruber, 1967, personal communication.

⁵ A. J. Kolombos, 1967, personal communication.